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Note

Concurrent TG–DTA measurements using a Cahn RG balance

W. W. WENDLANDT
Department of Chemistry, University of Houston, Houston, Texas 77004 (U.S.A.)
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The conversion of a DuPont Model 950 thermobalance for concurrent TG–DTA and TG–EC measurements has previously been described[1]. This was accomplished by using two outboard sample probes, one for DTA and the other for EC measurements. Two samples of the specimen were employed for the determination, one for TG and the other for DTA or EC. Even though the technique employed two samples, it had certain advantages over individual technique measurements[1].

Wendlandt[2] has also described a simple arrangement which permitted the actual sample temperature, T_s, to be recorded in TG measurements. Again, an outboard thermocouple was employed which required the use of two individual samples of the specimen to be used. We wish to extend this concept further to include concurrent TG–DTA measurements using a Cahn type electrobalance. This type of balance, due to its sensitive suspension, cannot contain sample and reference thermocouple wires attached to the balance beam, thus it is not possible to determine T_s and T_r on one sample.

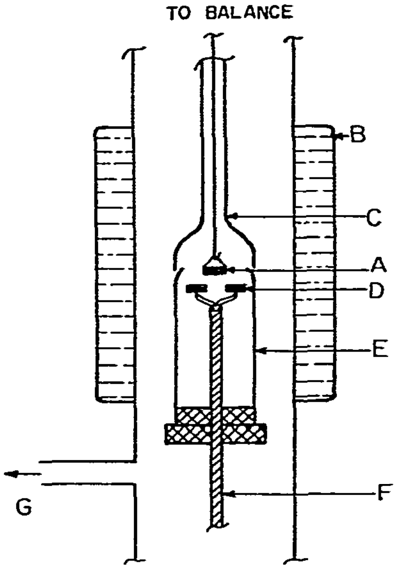
EXPERIMENTAL

TG–DTA apparatus

A schematic diagram of the furnace and sample holder is shown in Fig. 1.

The sample for TG measurements is placed in container A while that for DTA measurements is held in one of the identical containers at D. As in the DuPont thermobalance[1], the DTA probe is attached to the bottom of the furnace tube and can be easily removed for sample loading and removal. Considerable recorder curve noise is present unless the area around both the samples is properly baffled to prevent extravenous air currents in the furnace chamber. The TG sample is enclosed by baffle C, which is constructed of Pyrex glass, while the DTA baffle E is a tube made from nickel metal foil. For a controlled furnace atmosphere, such as dry nitrogen, gas flow is through the balance chamber, down the inside of baffles C and E, and out exit tube G.

The balance employed was a Cahn Model RG Electrobalance (not shown in



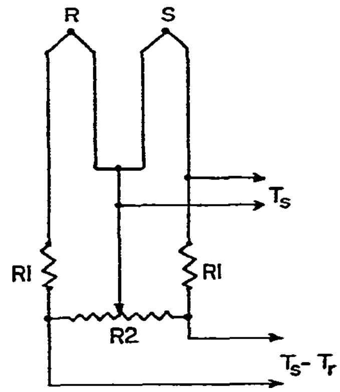


Fig. 1. Schematic diagram of furnace and sample holder for TG–DTA measurements. A, TG sample holder; B, furnace; C, TG baffle; D, DTA samples and reference holders; E, DTA baffle; F, ceramic support rod; and G, gas exit tube.

Fig. 2. DTA baseline compensation circuit. $R1 = 15\ \Omega$; $R2 = 200\ \Omega$, 10 turn potentiometer.

Fig. 1), although other similar balances could be used. A simple tube furnace was constructed from a Vycor tube (2.5 cm in diameter) wound with a Nichrome wire resistance element. The TG and DTA curves were multiplexed on a single *X–Y* plotter using a Donlee Laboratory electronic switch. Both functions were recorded versus the sample temperature, T_S, as detected by the DTA sample thermocouple.

DTA baseline compensator circuit

With a DTA apparatus such as this, there are always difficulties relating to the sloping baseline of the DTA curve. This problem was first noticed by Baxter[3] using the DuPont DSC cell and solved by employing a simple voltage divider circuit. This circuit was modified for use in this DTA apparatus, as shown in Fig. 2. The resistance values are not critical but should be within $\pm 50\%$ of the values cited. Adjustment of the 10 turn resistor, $R2$, will change the slope of the baseline either in a positive or negative direction. It is adjusted until the baseline remains flat over the temperature range of interest (usually 25 to 500°C).

ACKNOWLEDGMENT

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REFERENCES

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